

(12) **UK Patent Application** (19) **GB** (11) **2 248 091** (13) **A**
(43) Date of A publication 25.03.1992

(21) Application No 9118965.4

(22) Date of filing 04.09.1991

(30) Priority data

(31) 02253196

(32) 21.09.1990

(33) JP

(71) Applicant

Nissan Motor Company Limited

(Incorporated in Japan)

**No 2 Takara-cho, Kanagawa-ku, Yokohama-shi,
Kanagawa-ken, Japan**

(72) Inventors

Katsuhiko Kishi

Yoshiteru Yasuda

(74) Agent and/or Address for Service

Marks & Clerk

**57-60 Lincoln's Inn Fields, London, WC2A 3LS,
United Kingdom**

(51) INT CL⁵

F16C 33/12

(52) UK CL (Edition K)

F2A AD38 AD44 A114 A119

U1S S1842 S2319

(56) Documents cited

GB 1259672 A

GB 1114698 A

JP 03044436 A

US 3685878 A

(58) Field of search

UK CL (Edition K) F2A AD38 AD44

INT CL⁵ F16C 33/02 33/08 33/12

Online databases:WPI

(54) **Titanium based lubricating material**

(57) A titanium or titanium alloy substrate is subjected to an oxidation treatment before a solid lubricating film is applied to the substrate. The formed oxide can strengthen bonding force between the substrate and the solid lubricating film (e.g. MoS₂).

The resulting lubricating material is suitable for use in space or vacuum, light in weight, low in friction coefficient and long in life time.

GB 2 248 091 A

FIG. 1

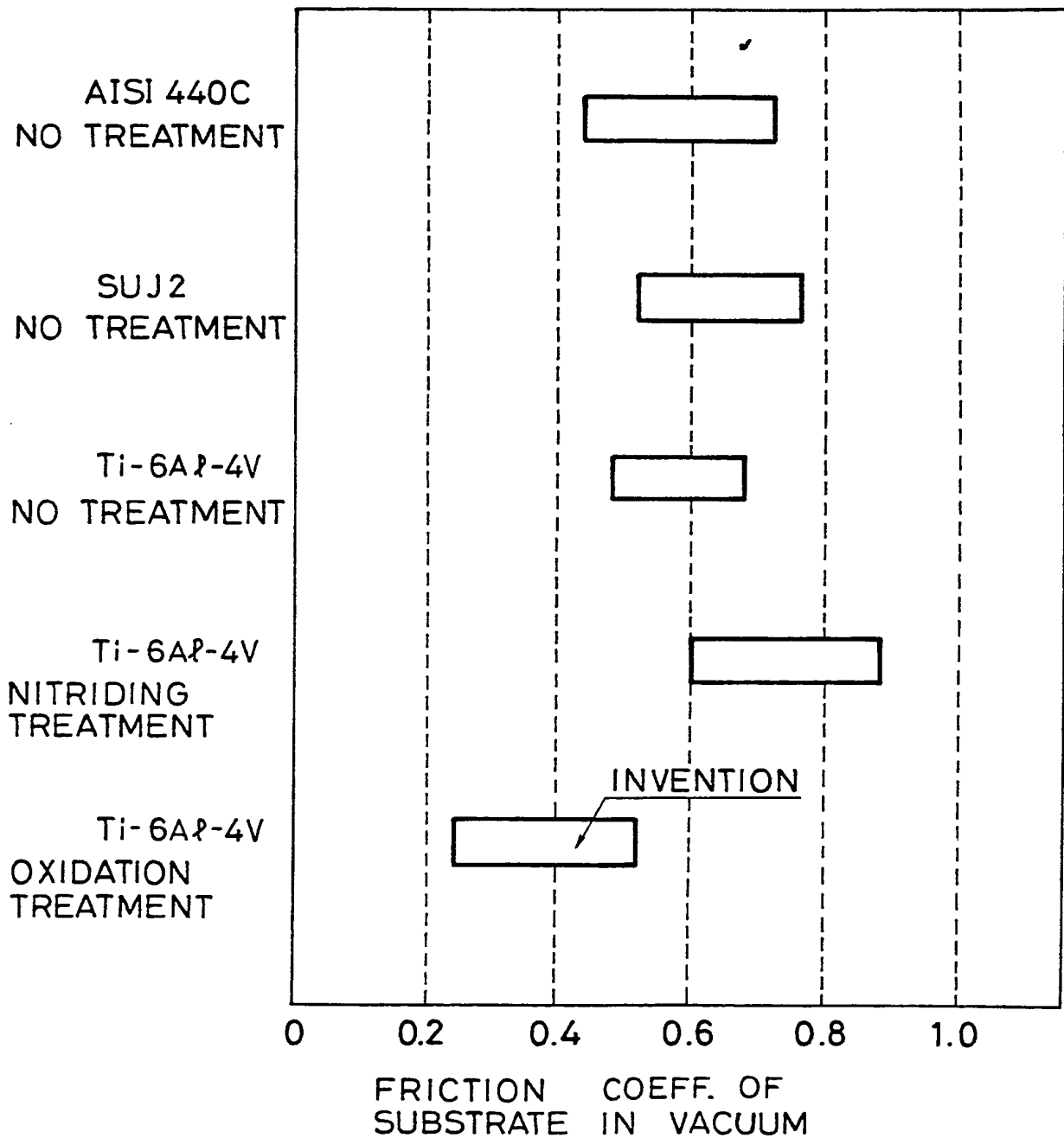


FIG. 2

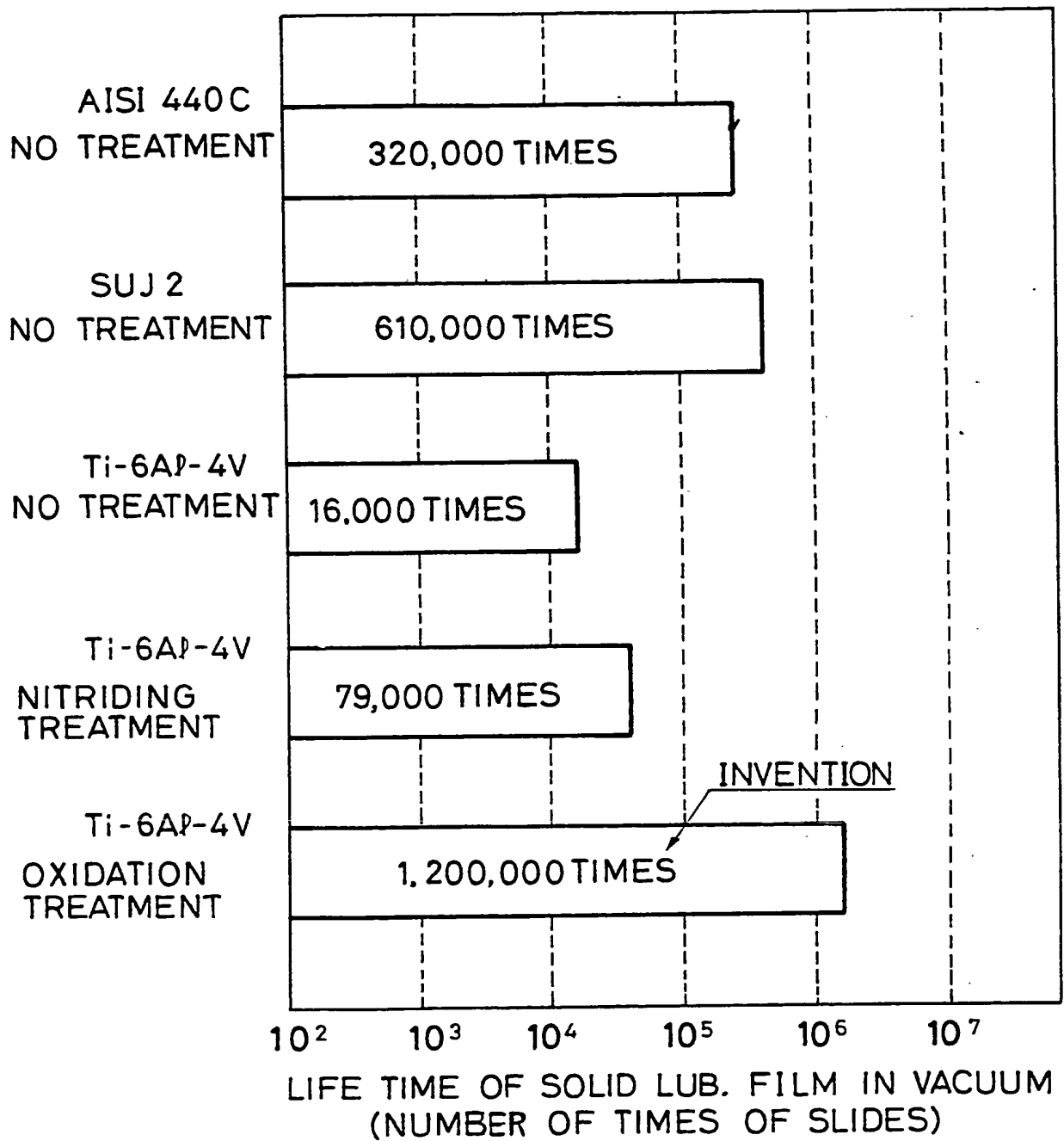
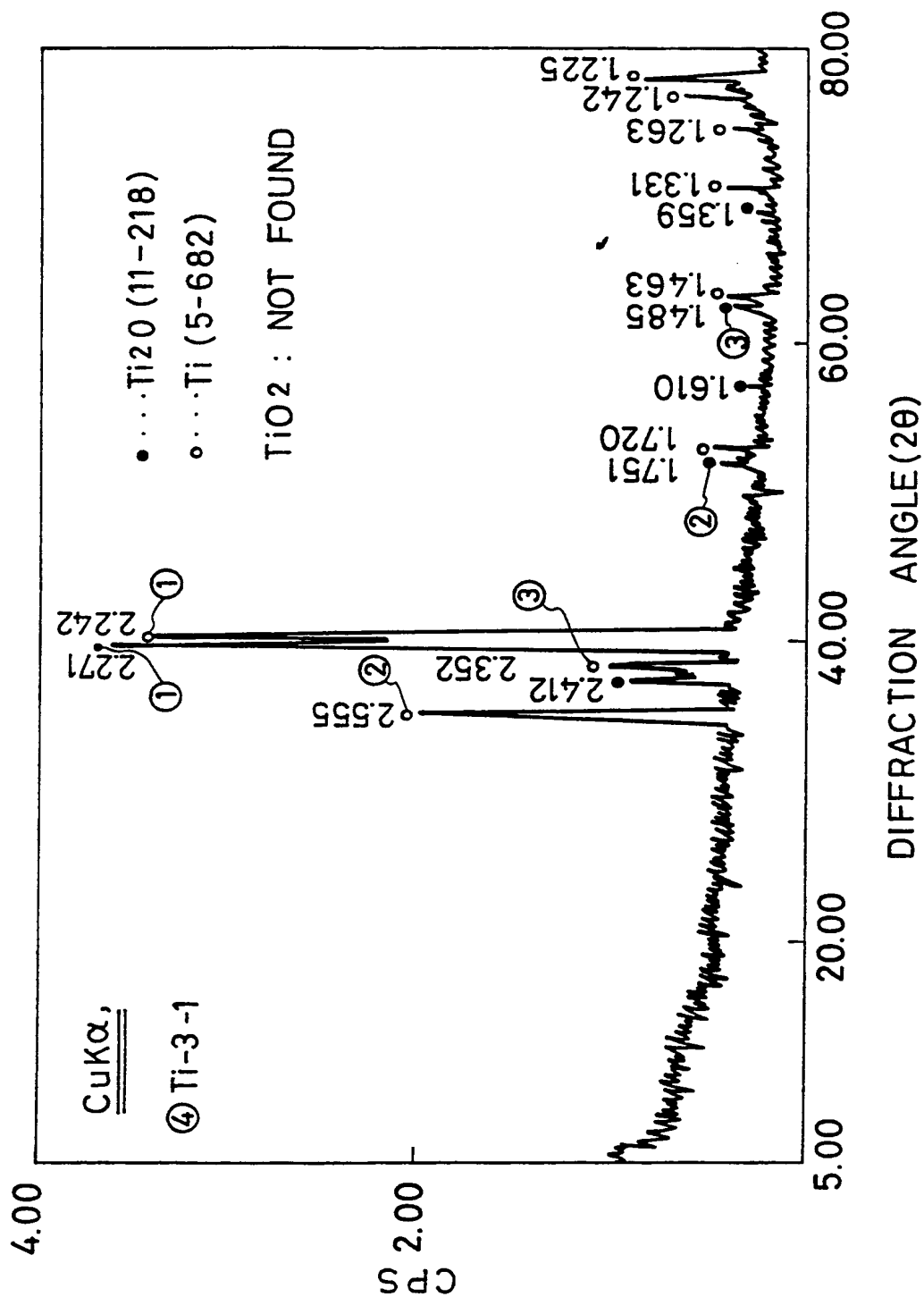


FIG. 3



TITANIUM LUBRICATING MATERIAL SUITABLE FOR USE IN VACUUM

The present invention relates to titanium lubricating material suitable for use in vacuum as light material for constructing a space station, in particular, and more specifically to a technology of improvement in surface quality of lubricating material usable for analysis instruments used in space or vacuum.

Conventionally there exist martensite-based stainless steels (e.g. AISI440C), high-chromium bearing steels (e.g. SUJ2), etc. as lubricating material suitable for use in vacuum.

To improve the surface quality of these substrates, the following technologies have been so far proposed:

(1) Nitriding onto the surface thereof (as disclosed in Japanese Published Unexamined (Kokai) Pat. Appli. No. 1-261561), and

(2) Ion implantation onto the surface thereof (as disclosed in Japanese Published Unexamined (Kokai) Pat. Appli. No. 1-205072).

In the prior-art lubricating material suitable for use in vacuum, since the substrate is made of iron, there exists a problem in that the weight thereof inevitably increases.

Further, when light alloy such as aluminium alloy or titanium alloy is used in such an environment, since the substrate is soft and therefore there exists a big difference in elastic deformation rate between a solid lubricating film and the substrate when used as lubricating material, there exists a problem in that bonding force is reduced at the boundary between the two and thereby the

life time of the solid lubricating film is shortened.

Therefore, there exists a need of development in lubricating material suitable for use in vacuum, which is light in weight and long in life time of the solid lubricating film.

Accordingly, it would be desirable to be able to provide a lubricating material suitable for use in vacuum, which is light in weight and long in life time of solid lubricating film, that is, excellent in durability.

In one aspect, the present invention provides a lubricating material usable in space or vacuum, comprising: (a) a substrate made of titanium or titanium alloy; and (b) a solid lubricating film applied to a surface of the substrate to which oxidation treatment has been effected. The principal component of the oxide formed on the substrate surface by the oxidation treatment is Ti_2O_3 . The oxidation treatment is effected by cleaning the substrate in hydrofluoric acid and then keeping the cleaned substrate at about $700 (\pm 20)^\circ C$ for 10 or more hours.

Further, the present invention provides a method of making a lubricating material usable in space or vacuum, comprising the steps of: (a) preparing a titanium or titanium alloy substrate; (b) cleaning the substrate; (c) keeping the cleaned substrate at about $700^\circ C$ for at least 10 hours for oxidation treatment; and (d) applying a solid lubricating film onto the substrate. Preferably: the titanium alloy includes 6 wt.% Al and 4 wt.% V; the substrate is cleaned by hydrofluoric acid; and the solid lubricating film is a $1\mu m$ -thick MoS_2 film, for instance.

In one titanium lubricating material suitable for use in vacuum according to the present invention, before a solid lubricating film is applied onto the substrate of titanium

or titanium alloy, since the substrate is cleaned by hydrofluoric acid, for instance, and held at 680 to 720°C for 10 or more hours within a high temperature furnace for oxidation treatment so that a thin oxide layer (T_2O) can be formed on the substrate surface to increase a bonding force between the substrate and the solid lubricating film, the weight of the material can be reduced and the life time thereof can be increased sufficiently.

The substrate obtained by the above-mentioned oxidation treatment is relatively low in frictional coefficient without lubricant in a vacuum. Further, when a solid lubricating film is formed on the substrate by applying a solid lubricating film, it has been found that the lubrication life time is longer than that of AISI 440C.

In the titanium lubricating material suitable for use in vacuum according to the present invention, since the substrate is made of titanium or titanium alloy and additionally oxidation treatment is effected on the substrate surface before a solid lubricating film is applied onto the substrate surface, the oxide layer formed by the oxidation treatment can strengthen the bonding force between the substrate and the solid lubricating film, thus improving the life time of the solid lubricating film.

The invention will be described further, by way of example, with reference to the accompanying drawings, in which:

Fig. 1 is a graphical representation showing the test results of dynamic friction coefficients of various test samples in a vacuum;

Fig. 2 is a graphical representation showing the test results of life time of solid lubricating film applied onto the surfaces of various test samples, in a vacuum; and

Fig. 3 is a graphical representation showing the analysis results of oxide formed on the substrate to which oxidation treatment has been effected according to the present invention, which are analyzed in accordance with X-

ray diffraction technique.

Embodiments of the present invention will be explained hereinbelow.

Frictional Coefficient under Non-lubrication

Sample material for use in the invention was obtained as follows:

Titanium alloy including 6 wt.% Al and 4 wt.% V was selected as the substrate; the selected substrate was cleaned in hydrofluoric acid and then held at 720°C for 16 hours within a high temperature furnace in the atmosphere for oxidation treatment so that a thin oxide layer was formed on the surface of the substrate.

For comparison, the following comparative samples were also prepared:

Titanium alloy including 6 wt.% Al and 4 wt.% V was held at 750°C for 20 hours in N₂ gas for nitriding treatment so that a thin nitride layer was formed on the surface of the substrate.

Titanium alloy including 6 wt.% Al and 4 wt.% V without any surface treatment was prepared; SUJ 2 bearing steel without any surface treatment was prepared; and AISI 440C martensite-based stainless steel without any surface treatment was prepared. The above-mentioned samples are listed in Table 1 below.

Table 1

Substrate	Surface treatment	Surface treatment conditions
AISI 440C	None	_____
SUJ 2	None	_____
Ti-6Al-4V	None	_____
Ti-6Al-4V	Nitriding	at 750°C for 20 hrs in N ₂ gas
Ti-6Al-4V	Oxidation (Invention)	at 700°C for 16 hrs in atm.

The above five samples were tested to check dynamic friction characteristics under no-lubrication conditions in a vacuum in accordance with a pin-disk test machine in which a pin is brought into pressure contact with the surface of a rotating disk formed of each of these samples under the test conditions as listed in Table 2 below.

Table 2

Pin	AISI 440C 5/16 inch (8mm) ball
Load	2N (about 200g)
Speed	0.5m/s
Atm.	Vacuum (5×10^{-7} Torr or less)
Temp.	Room temp.

Fig. 1 shows the test results of the above dynamic friction characteristics of these samples. Fig. 1 indicates that the dynamic friction coefficient of the sample for use in the present invention (i.e. Ti-6% Al-4% V with oxidation treatment) is clearly lower than those of the other samples.

Life Time of Solid Lubricating Film

The life times (the number of times of sliding motions) of a solid lubricating film applied onto the

substrate surfaces of the same test samples listed in Table 1, respectively were tested with the same pin-disk test machine under the test conditions as listed in Table 3 below. A $1\mu\text{m}$ -thick MoS_2 (molybdenum disulfide) film was formed as the solid lubricating film onto each sample surface with a high-frequency sputtering apparatus.

Table 3

Pin	AISI 440C 5/16 inch ball
Load	10N (about 1kg)
Speed	0.5m/s
Atm.	Vacuum (5×10^{-7} Torr or less)
Temp.	Room temp.

Fig. 2 shows the test results of the above life times of the solid lubricating film (MoS_2) applied onto these test samples. Fig. 2 indicates that the life time of the solid lubricating film applied onto the substrate to which oxidation treatment is effected is remarkably longer than those of other samples, that is, the same substrates to which no surface treatment is effected or nitriding treatment is effected or other material such as AISI 440C or SUJ 2.

Analyzed Results of Oxide formed on Sample Surface

Fig. 3 shows the analyzed results of the surface oxidization substance of the substrate to which oxidation treatment is effected according to the present invention, in accordance with X-ray diffraction technique.

Fig. 3 indicates that the oxide formed on the substrate surface is not TiO_2 but Ti_2O , which is an unstable oxide. Therefore, it is possible to consider that the formation of Ti_2O can markedly improve the bonding force between the substrate and the lubrication film. The mutual action between this surface oxide and the solid

lubricating film is not yet well clarified; however, it may be due to the fact that some surface chemical reaction increases the bonding force between the lowermost layer of the solid lubricating film and the substrate and therefore
5 the life time of the solid lubricating film can be improved.

In the titanium lubricating material suitable for use in vacuum according to the present invention, since oxidation treatment is effected to the surface of the
10 substrate of titanium or titanium alloy before a solid lubricating film is applied, it is possible to provide a titanium substrate member small in dynamic friction coefficient in space or vacuum environment and long in solid lubricating film life time as compared with the
15 conventional martensite-based stainless steels or bearing steels. In addition, since titanium or titanium alloy is used as the substrate, it is possible to reduce the weight thereof by about 43% as compared with the conventional iron substrate. Therefore, the material of the present
20 invention is preferably usable not only for bearings of vacuum equipments such as electron microscopes, physical vapour deposition (PVD) apparatus, etc., but also lubricating members for space instruments which require light material in particular.

Claims:-

1. A lubricating material for use in a space or in a vacuum, comprising:
 - (a) a substrate made of titanium or titanium alloy; and
 - (b) a solid lubricating film applied to an oxidized surface of the substrate.
2. A lubricating material as claimed in claim 1, wherein the principal component of the oxidized substrate surface is Ti_2O_3 .
3. A lubricating material as claimed in claim 1 or 2, wherein the oxidized substrate surface has been obtained by cleaning the substrate in hydrofluoric acid and then keeping the cleaned substrate at about $700^{\circ}C$ in an oxidizing atmosphere for at least 10 hours.
4. A lubricating material as claimed in any preceding claim, wherein the titanium alloy includes 6 wt.% Al and 4 wt.% V.
5. A lubricating material as claimed in any preceding claim, wherein the solid lubricating film is an MoS_2 film.
6. A lubricating material as claimed in any preceding claim, wherein the solid lubricating film is $1\mu m$ thick.
7. A method of making a lubricating material usable in space or in a vacuum, comprising:
 - cleaning a titanium or titanium alloy substrate;
 - keeping the cleaned substrate at about $700^{\circ}C$ in an oxidizing atmosphere for at least 10 hours for oxidation treatment; and
 - then applying a solid lubricating film to the substrate.

8. A method as claimed in claim 7, wherein the oxide formed on the substrate by the oxidation treatment is Ti_2O_3 .
9. A method as claimed in claim 7 or 8, wherein the titanium alloy includes 6 wt.% Al and 4 wt.% V.
10. A method as claimed in any of claims 7 to 9, wherein the substrate is cleaned by hydrofluoric acid.
11. A method as claimed in any of claims 7 to 10, wherein the solid lubricating film is an MoS_2 film.
12. A method as claimed in any of claims 7 to 11, wherein the solid lubricating film is 1 μm thick.
13. A method as claimed in claim 7, substantially as described herein with reference to the accompanying drawings.
14. A lubricating material obtained by a method according to any of claims 7 to 13.
15. A lubricating material as claimed in any of claims 1 to 6 or claim 14, when used in space or in a vacuum.



THIS PAGE BLANK (USPTO)